

FORMULATION GUIDE OF EXILVA WITH ALCOHOLS

This document contains laboratory formulation examples/guides of Exilva and different concentrations of Ethanol and Isopropanol.

Exilva grades are usually available as:

- a “ready to use” dispersion with around 2wt% of fibrils in water (the L concentration)
- a paste with around 10wt% of fibrils in water (the V concentration)

In this study, all tests are carried out with the F grade. Please note that preserved Exilva cosmetic grades are available when required.

Exilva products tested:

- Exilva F 01-L
- Exilva F 01-V

The initial laboratory experiments show that around 0,5wt% of Exilva MicroFibrillated Cellulose MFC (dry base) is required to get a stable and homogeneous formulation over time.

In order to add 0,5wt% MFC into a formulation, the following weight concentration of the Exilva products in the total end formulation is required:

- 25wt% of Exilva F 01-L
- 5wt% of Exilva F 01-V

When possible to replace around 20 to 25wt% of free water from the formulation, Exilva F 01-L is the recommended product to test. If not, Exilva F 01-V is the recommended product. High shear is then required.



FIGURE 1: Exilva V-grade (10wt%)



FIGURE 2: Exilva L-grade (2wt%)

THIS DOCUMENT CONTAINS THE FOLLOWING:

1. Summary of how to formulate with Exilva F 01-L and Exilva F 01-V
2. Formulation of Exilva F 01-L with 60% Ethanol
3. Formulation of Exilva F 01-L with 60% Ethanol, without denaturant
4. Formulation of Exilva F 01-L with 70% Ethanol
5. Formulation of Exilva F 01-V with 70% Ethanol
6. Formulation of Exilva F 01-V with 80% Ethanol
7. Formulation of 5wt% Exilva F 01-V with 90% Ethanol
8. Formulation of 7wt% Exilva F 01-V with 90% Ethanol
9. Formulation of Exilva F 01-V with 80% Isopropanol
10. Annex: Percentage by volume (v/v) versus Percentage by weight (wt%)

1. SUMMARY OF HOW TO FORMULATE WITH EXILVA F 01-L AND EXILVA F 01-V

	EXILVA F 01-L	EXILVA F 01-V
<i>Recommended start dosage:</i>	<p>Start by adding 25wt% of product as delivered. If you add 25wt% of product as delivered, this corresponds to 0,5wt% MFC and 24,5wt% water.</p> <p>Replace 25wt% of your free water with Exilva F 01-L if possible.</p>	<p>Start by adding 5wt% of product as delivered. If you add 5wt% of product as delivered, this corresponds to 0,5wt% MFC and 4,5wt% water.</p> <p>Replace 5wt% of your free water with Exilva F 01-V if possible.</p>
<i>Equipment and way of dispersing:</i>	<p>Exilva F 01-L can be introduced to the formulation using overhead stirrers under high or low shear.</p> <p>Recommended to use high shear when possible. Avoid foam formation (do not use high shear after adding foaming ingredients)</p>	<p>For successful dispersion of Exilva F 01-V, high shear is required during or after the addition of Exilva.</p> <p>A dissolver or rotor-stator type mixer with tip speed above 6 m/s with 15 min mixing time is recommended.</p>

TABLE 1: Summarized differences when handling Exilva L-grade and V-grade.

	EXILVA F 01-L	EXILVA F 01-V
SOLID CONTENT	2wt%	10wt%
RECOMMENDED START DOSAGE (% AS IS) AS PART OF FINAL FORMULATION	25wt%	5wt%
<i>CORRESPONDS TO FIBRILS</i>	0,5wt%	0,5wt%
<i>CORRESPONDS TO WATER</i>	24,5wt%	4,5wt%
WATER TO REPLACE WITH EXILVA	25wt%	5wt%
EQUIPMENT	OVERHEAD STIRRER, DISSOLVER, ROTOR-STATOR	DISSOLVER, ROTOR-STATOR
DISPERSION	(LOW /) HIGH SHEAR	HIGH SHEAR

IMPORTANT WHEN TESTING EXILVA:

- ✓ Recommended to test directly in final system and not only in water
- ✓ Avoid foam formation. Foam formation during addition or mixing of Exilva will lead to phase separation.

ALWAYS EXPLORE:

1. The addition order of Exilva into the formulation
 - Exilva is to be added to most polar phase
 - Non-polar solvents are to be added to dispersed Exilva slowly and under stirring.
2. The shear applied when incorporating Exilva
 - High shear is always recommended when possible
 - Be careful and avoid foam formation when using high shear mixing.

2. FORMULATION OF EXILVA F 01-L WITH 60% ETHANOL

Sanitizing Formulation: 60% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-20

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	WATER (ION-EXCHANGED)	WATER	13,55	SOLVENT	-
2	GLYCEROL (98%)	GLYCERIN	1,45	DENATURANT, HUMECTANT	PROLAB
3	EXILVA F 01-L (2wt%)*	WATER; CELLULOSE	25,00	RHEOLOGY ADDITIVE	BORREGAARD
4	ETHANOL (>99%)	ALCOHOL	60,00	ACTIVE	ANTIBAC AS

**Exilva F 01-L is a 2wt% dispersion of Microfibrillated Cellulose (MFC) in water. Using 25wt% of Exilva F 01-L in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 24,5wt% water.*

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. Now pour ingredient 3 into the blend of 1 and 2 and stir using an overhead stirrer until homogeneous.
3. While under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
4. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 100 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

DISCLAIMER: The suggested formula is for your convenience and testing. Under your production conditions, it may show other values or characteristics. No warranty or representation of any kind is made, given or implied as to the sufficiency or fitness for purpose nor as to the absence of any infringement of any proprietary rights of third parties.

3. FORMULATION OF EXILVA F 01-L WITH 60% ETHANOL, WITHOUT DENATURANT

Sanitizing Formulation: 60% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-19

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	WATER (ION-EXCHANGED)	WATER	15,00	SOLVENT	
2	EXILVA F 01-L (2wt%)*	WATER; CELLULOSE	25,00	RHEOLOGY ADDITIVE	BORREGAARD
3	ETHANOL (>99%)	ALCOHOL	60,00	ACTIVE	ANTIBAC AS

* Exilva F 01-L is a 2wt% dispersion of Microfibrillated Cellulose (MFC) in water. Using 25wt% of Exilva F 01-L in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 24,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. While under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
3. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 100 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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4. FORMULATION OF EXILVA F 01-L WITH 70% ETHANOL

Sanitizing Formulation: 70% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-17

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	GLYCEROL (98%)	GLYCERIN	5,00	DENATURANT, HUMECTANT	PROLAB
2	EXILVA F 01-L (2wt%)*	WATER; CELLULOSE	25,00	RHEOLOGY ADDITIVE	BORREGAARD
3	ETHANOL (>99%)	ALCOHOL	70,00	ACTIVE	ANTIBAC AS

* Exilva F 01-L is a 2wt% dispersion of Microfibrillated Cellulose (MFC) in water. Using 25wt% of Exilva F 01-L in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 24,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. While under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
3. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 200 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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5. FORMULATION OF EXILVA F 01-V WITH 70% ETHANOL

Sanitizing Formulation: 70% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-10

#	INGREDIENTS	INCI	%WT	FUNCTION	SUPPLIER
1	WATER (ION-EXCHANGED)	WATER	20,00	SOLVENT	
2	GLYCEROL (98%)	GLYCERIN	5,00	DENATURANT, HUMECTANT	PROLAB
3	EXILVA F 01-V (10wt%)*	WATER; CELLULOSE	5,00	RHEOLOGY ADDITIVE	BORREGAARD
4	ETHANOL (>99%)	ALCOHOL	70,00	ACTIVE	ANTIBAC AS

* Exilva F 01-V is a 10wt% paste of Microfibrillated Cellulose (MFC) in water. Using 5wt% of Exilva F 01- V in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 4,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. Now pour ingredient 3 into the blend of 1 and 2 and stir using a dissolver or rotor-stator type mixer that can provide high shear to disperse the Exilva paste into this blend until the blend is homogeneous. Ultraturrax mixer is used in this step at 20 000 rpm for 4 minutes.
3. While still under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
4. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 300 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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6. FORMULATION OF EXILVA F 01-V WITH 80% ETHANOL

Sanitizing Formulation: 80% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-05

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	WATER (ION-EXCHANGED)	WATER	10,00	SOLVENT	
2	GLYCEROL (98%)	GLYCERIN	5,00	DENATURANT, HUMECTANT	PROLAB
3	EXILVA F 01-V (10wt%)*	WATER; CELLULOSE	5,00	RHEOLOGY ADDITIVE	BORREGAARD
4	ETHANOL (>99%)	ALCOHOL	80,00	ACTIVE	ANTIBAC AS

* Exilva F 01-V is a 10wt% paste of Microfibrillated Cellulose (MFC) in water. Using 5wt% of Exilva F 01- V in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 4,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. Now pour ingredient 3 into the blend of 1 and 2 and stir using a dissolver or rotor-stator type mixer that can provide high shear to disperse the Exilva paste into this blend until the blend is homogeneous. Ultraturrax mixer is used in this step at 20 000 rpm for 4 minutes.
3. While still under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
4. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 300 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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7. FORMULATION OF 5 WT% EXILVA F 01-V WITH 90% ETHANOL

Sanitizing Formulation: 90% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-04

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	GLYCEROL (98%)	GLYCERIN	5,00	DENATURANT, HUMECTANT	PROLAB
2	EXILVA F 01-V (10wt%)*	WATER; CELLULOSE	5,00	RHEOLOGY ADDITIVE	BORREGAARD
3	ETHANOL (>99%)	ALCOHOL	90,00	ACTIVE	ANTIBAC AS

* Exilva F 01-V is a 10wt% paste of Microfibrillated Cellulose (MFC) in water. Using 5wt% of Exilva F 01-V in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 4,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using a dissolver or rotor-stator type mixer that can provide high shear to disperse the Exilva paste into this blend until the blend is homogeneous. Ultraturrax mixer is used in this step at 20 000 rpm for 4 minutes.
2. While still under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
3. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 800 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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8. FORMULATION OF 7 WT% EXILVA F 01-V WITH 90% ETHANOL

Sanitizing Formulation: 90% Ethanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-07

#	INGREDIENTS	INCI	% WT	FUNCTION	SUPPLIER
1	GLYCEROL (98%)	GLYCERIN	3,00	DENATURANT, HUMECTANT	PROLAB
2	EXILVA F 01-V (10wt%)*	WATER; CELLULOSE	7,00	RHEOLOGY ADDITIVE	BORREGAARD
3	ETHANOL (>99%)	ALCOHOL	90,00	ACTIVE	ANTIBAC AS

* Exilva F 01-V is a 10wt% paste of Microfibrillated Cellulose (MFC) in water. Using 7wt% of Exilva F 01-V in a formulation corresponds to the use of 0,7wt% MFC in the final formulation and 6,3wt% water

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using a dissolver or rotor-stator type mixer that can provide high shear to disperse the Exilva paste into this blend until the blend is homogeneous. Ultraturrax mixer is used in this step at 20 000 rpm for 4 minutes.
2. While still under stirring, add the Ethanol very slowly to the blend while mixing continuously. If you do not add the Ethanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
3. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1300 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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9. FORMULATION OF EXILVA F 01-V WITH 80% ISOPROPANOL

Sanitizing Formulation: 80% Isopropanol, Possibility to Spray

COMPOSITION:

Sample ID: 042020-18

#	INGREDIENTS	INCI	%WT	FUNCTION	SUPPLIER
1	WATER (ION-EXCHANGED)	WATER	10,00	SOLVENT	
2	GLYCEROL (98%)	GLYCERIN	5,00	DENATURANT, HUMECTANT	PROLAB
3	EXILVA F 01-V (10wt%)*	WATER; CELLULOSE	5,00	RHEOLOGY ADDITIVE	BORREGAARD
4	ISOPROPANOL (> 98%)	ISOPROPYL ALCOHOL	80,00	ACTIVE	VWR

* Exilva F 01-V is a 10wt% paste of Microfibrillated Cellulose (MFC) in water. Using 5wt% of Exilva F 01- V in a formulation corresponds to the use of 0,5wt% MFC in the final formulation and 4,5wt% water.

PROCEDURE:

Lab batch size: 250g.

1. Pour ingredients 1 and 2 into the container and stir using an overhead stirrer until homogeneous.
2. Now pour ingredient 3 into the blend of 1 and 2 and stir using a dissolver or rotor-stator type mixer that can provide high shear to disperse the Exilva paste into this blend until the blend is homogeneous. Ultraturrax mixer is used in this step at 20 000 rpm for 4 minutes.
3. While still under stirring, add the Isopropanol very slowly to the blend while mixing continuously. If you do not add the Isopropanol slowly into the Exilva dispersion, you will feel the fibrils. Slow addition will maintain the Exilva structure and avoid feeling any residues.
4. Formulation is ready to use.

SPECIFICATION VALUES:

Appearance: Homogeneous non-transparent and opaque formulation

Feel: Non-tacky non-sticky skin feel.

Sprayability: Sprayable with a non-dripping effect

Viscosity: Brookfield viscosity measured: Approx. 1 200 mPa s

Brookfield Viscosity measured after 24hrs and 1 week from sample preparation using Brookfield RVDII viscometer:

- 200 mL sample in a 250 mL high beaker
- Spindle V-72
- Measure the viscosity at 10 rpm for 5 min.
Use the last value as viscosity.

STABILITY

- Appearance and Brookfield viscosity stable for at least 1 week at 20°C.
- Stability still ongoing.

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10. ANNEX: PERCENTAGE BY VOLUME (V/V) VS. PERCENTAGE BY WEIGHT (WT%)

Concentrations can be expressed as percentage by volume (v/v) and percentage by weight (wt%). Note that all formulations in this document are reported in percentage by weight (wt%).

Since the density of Exilva products is around 1g/mL, recommended start dosage of the Exilva products in percentage by volume are:

- 25 v/v % of Exilva F 01-L
- 5 v/v% of Exilva F 01-V

The density of Glycerin is 1,25 g/mL.

The density of Ethanol is 0,789 g/mL and the density of Isopropanol is 0,786 g/mL.

Note that the Ethanol contracts when mixed with water.

V/V VS. WT%, ETHANOL

For ethanol, below is a table that allows for the most commonly used concentrations to compare the values of both scales.

CONCENTRATION OF ETHANOL BY WEIGHT (W/W)	CONCENTRATION OF ETHANOL BY VOLUME (V/V)
60,0% (W/W)	67,7% (V/V)
70,0% (W/W)	77,0% (V/V)
80,0% (W/W)	85,5% (V/V)
90,0% (W/W)	93,3% (V/V)

Reference: *European Pharmacopoeia 7.0, 2011.*